Verification of Reference Materials

1 Purpose

This document sets forth the procedures regarding storage and verification of reference materials and supplements the requirements in the FBI Laboratory *Quality Assurance Manual (QAM)* and the FBI Laboratory *Operations Manual (LOM)*. These procedures are to be used in conjunction with the Performance Monitoring Protocols (PMPs) for individual instruments and the Standard Operating Procedures (SOPs) for analysis of evidence.

2 Scope

These procedures apply to case working personnel conducting work in explosives chemistry analysis who use reference materials in casework.

3 Equipment/Materials/Reagents

The equipment, materials, and reagents used to verify a reference material will depend upon the nature (e.g., organic/inorganic, polar/nonpolar, solid/liquid) of the substance. Most of these verifications will be performed following an analysis SOP. The equipment, materials, and reagents required for such verifications will be listed within the SOP used as well as in the Explosives Instrument Parameters and Reagent Preparation SOP.

4 Standards and Controls

Standards and controls referenced in this document are acquired, purchased, or synthesized.

5 Definitions

5.1 Reference Material

A material with known origin, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

5.2 Certified Reference Material

Reference material, characterized by a metrologically valid procedure for one or more specified properties, accompanied by a reference material certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

6 Procedures

6.1 Opening a New Reference Material

When opening reference materials for the first time, it is good laboratory practice to record the following information directly on the container:

- Date opened
- Initials of the person opening the container
- Expiration date, if applicable

6.2 Synthesis of a Reference Material

When a suitable reference material is not available from a vendor it may be necessary to synthesize it (e.g., peroxide based explosives). The following information will be recorded and maintained in the validation file:

- Name of synthesized reference material
- Procedure or notes used to synthesize the material
- Date of synthesis
- Initials of the person who synthesized the material
- Lot number, if applicable
- Storage instructions, if applicable
- Verification of identity data

When used in casework, cite the database's unique identifier or record the following information in the examination records for the case in which it was used:

- Name of synthesized reference material
- Date synthesized
- Lot number, if applicable

Note that detailed synthesis procedures, notes, and/or recipes of explosives may be considered "Law Enforcement Sensitive." Refer to applicable classification guides for proper marking of this information.

6.3 Verification of a Reference Material

All chemical reference materials must have their identities verified prior to, or in concurrence with, use in casework. Certified reference materials do not require further verification. For all other reference materials, only one sample per manufacturer's lot number of the reference material must be verified prior to use. Subsequent reference materials from the same lot will be considered as having the same verification as the original.

If the reference material will be used for quantitative work, then the purity of the reference material must also be verified prior to, or in concurrence with casework. The following lists the steps necessary to perform verifications:

6.3.1 Identity Verification (Qualitative)

The following techniques can be used to verify the identity of the reference material:

- Fourier Transform Infrared Spectroscopy (FTIR)
- Gas Chromatography with Mass Spectrometry (GC/MS)
- Gas Chromatography (GC) with electron capture (ECD) or flame ionization detectors (FID)
- High Performance Liquid Chromatography (HPLC) with applicable detector(s)
- High Resolution Mass Spectrometry (e.g., OrbiTrap)
- Ion Chromatography (IC) with conductivity detector
- Ion Chromatography with Mass Spectrometry (IC/MS)
- Liquid Chromatography with Mass Spectrometry (LC/MS)
- Raman spectroscopy
- Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM/EDS)
- Solids Probe Mass Spectrometry (SP/MS)
- Ultra Performance Liquid Chromatography with Mass Spectrometry (UPLC/MS)
- X-ray diffraction (XRD)

For each instrumental technique, refer to the appropriate PMP and the Explosives Instrument Parameters and Reagent Preparation SOP for instrument usage procedures, parameters, and reagent preparation information. Prior to sample analysis, the PMP for the instrument must be followed to conduct a Quality Assurance/Quality Control (QA/QC) check to verify the instrument's reliability and reproducibility from analysis to analysis. Instrumentation not listed above may be used, provided it is shown to be in proper working order prior to use.

Reference materials supplied with a Certificate of Analysis (or equivalent) will be verified by at least one technique. Results must compare favorably with a previously analyzed reference material, reference data, or literature, as necessary.

Synthesized reference materials and reference materials not supplied with a Certificate of Analysis (or equivalent) will be verified by at least two techniques (including at least one spectroscopic technique). Results must compare favorably with a previously analyzed reference material, reference data, or literature, as necessary.

When the identity verification is complete, the data will be maintained on the respective instruments, in the case file, or be printed and filed in the validation file.

Example: A GC/ECD testmix component of nitroglycerin (with a Certificate of Analysis) purchased from a new vendor will be verified by an instrumental technique such as GC/ECD. It may then be used as a reference material to compare against an unknown evidence item.

Example: A reagent grade chemical of strontium nitrate (without a Certificate of Analysis) is purchased for use as a reference material for the very first time. It will be analyzed by two techniques (including at least one spectroscopic technique) such as FTIR and XRD to verify its identity prior to being used as a reference material to compare against an unknown evidence item.

6.3.2 Purity Verification (Quantitative)

If an item needs to be quantitated, the following steps will be used to verify the purity of the reference material. Verification of the purity of a reference material will be performed after the identity verification and prior to quantitative use.

A variety of techniques may be used to confirm the purity/concentration of the reference material. Only one technique is needed. Acceptable techniques for purity verification include:

- Gas Chromatography with applicable detector(s)
- Liquid Chromatography with applicable detector(s)

Prior to use, verify that the instrument is in proper working order by following the instrument's PMP. When the purity verification is completed, the applicable data and instrumental parameters will be printed and filed in the validation file.

6.3.2.1 Gas Chromatography (GC) and Liquid Chromatography (LC)

An individual will:

- Accurately dilute the reference material to an appropriate concentration in an appropriate solvent (e.g., 100 ppm for GC/MS and 1-10 ppm for LC/MS and HPLC).
- Accurately dilute a previously verified reference material of the same analyte, a previously calibrated deuterated analog, or a reference material of the same

analyte from a different lot, to the same concentration in the same solvent as used for the new reference material.

- Analyze the new diluted reference material solution with appropriate instrumental parameters. The file parameters should include the name of the supplier, the lot number of the reference material, and your initials. The analysis should be performed at least three times and the average area of the peak obtained.
- Following the same instrumental parameters, analyze the previously verified material solution, the deuterated material solution, or the solution of the reference material from a different lot. (Optionally, a calibration curve of multiple points of the older reference material may be used to determine the purity of the new reference material.)
- Comparison of the average areas from the two reference materials allows for calculation of the concentration (and thus the purity) of the new reference material, taking into account a degree of analytical error.
- Print the applicable data and the instrumental parameters and file in the validation file.

6.4 Reference Material Verification Discrepancies

6.4.1 Discrepancies in Identity

Discrepancies in the structural identity of a reference material following qualitative testing will be discussed with the manufacturer and, if necessary, actions will be taken to obtain another reference material. If the material is retained, the container will be labeled with information indicating the discrepancy.

6.4.2 Discrepancies in Purity

It should be noted that the use of quantitative positive controls are effective in verifying the concentration of a reference material. The use of such controls allow for the verification of the stability of a reference material after the initial qualitative testing.

If the purity verification of a reference material results in an average purity within ±5% of the listed purity, the listed purity will be used in preparing future calibrators or controls from this reference material. Discrepancies in the purity of a reference material greater than ±5% of the listed purity will result in the assignment of an approximate purity value (or concentration) to the material. This new purity (or concentration) will be recorded on the container and used in preparing future calibrators or controls from this reference material.

6.5 Reverification of Reference Materials

The expiration date for reference materials is determined by the expiration date provided by the manufacturer or determined by the individual SOP (or other applicable document) describing its

preparation. Reference materials may be reverified and have their expiration dates extended by the initial expiration timeframe or used past their expiration dates provided that appropriate steps are taken with every use to demonstrate and re-verify their reliability.

6.6 Use of Reference Materials

Reference materials may be used for analysis or comparison in casework and/or evaluation of instrumentation and equipment.

At the time of use, include the relevant information from the label or cite the database's unique identifier in the examination records.

6.7 Storage of Reference Materials

The reference material will be stored following manufacturer's recommendations.

Reference materials that have been diluted in a solvent will be stored in a refrigerator unless indicated otherwise by the manufacturer or PMP.

Dry chemicals will be stored according to the manufacturer recommendations, if possible.

All reference materials should be stored in a central location and made available to others in the unit.

6.8 Transportation of Reference Materials

When a reference material (which is deemed a Department of Transportation (DOT) hazardous material), is shipped or transported outside the FBI Laboratory, the preparer will ensure that the material is packaged in accordance with DOT shipping regulations, as appropriate. When a commercial shipper is used, such as FedEx, the individual responsible for packaging for the reference material will comply with all regulations set forth by DOT and the commercial shipping company, as appropriate. When a reference material is transported by FBI vehicle or aircraft, the material will be packaged appropriately to prevent the possibility of breakage, contamination, or other alterations to the material.

6.9 Reference Material Records

All Certificates of Analysis (or equivalent) will be stored in the validation file. Qualitative verification data, purity verification data, and any other records necessary to properly characterize the reference material will be stored in the validation file.

7 Limitations

Limitations may be specific to a particular reference material (e.g., if the reference material has an expiration date, if the manufacturer changes the formulation of their product).

The limitations associated with these procedures are dependent on the instrumental techniques used to determine the identity and purity of reference materials. In general, the listed techniques will be sufficient for these determinations when performed as described.

8 Safety

Safety protocols, contained within the FBI Laboratory Safety Manual, will be observed at all times. This manual also contains information on the proper handling and disposal of all chemicals.

Refer to the PMP for the specific instrument for additional safety information. Standard precautions will be taken for the handling of all chemicals, reagents, and standards. Some of the chemicals may be carcinogenic. Personal protective equipment will be used when handling any chemical and when performing any type of analysis.

The handling of some explosive materials is hazardous due to potential ignition by heat, shock, friction, impact, or electrostatic discharge. Personnel should work with small quantities of material (such as a few grams) and properly store larger quantities in approved containers.

9 References

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

FBI Laboratory Safety Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

Explosives Standard Operating Procedures: Chemistry, Federal Bureau of Investigation, Laboratory Division, latest revisions.

Instrument Operations Manuals for the specific models and accessories used.

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Pfleger, K., Maurer, H., Weber, A., *Mass Spectral and GC Data of Drugs, Poisons, Pesticides, Pollutants, and Their Metabolites,* 2nd ed., VCH: Weinheim, Germany, 1992.

Pouchert, C. J., *The Aldrich Library of Infrared Spectra* 2nd ed., Aldrich Chemical Co., 1975.

Rev. #	Issue Date	History
6	12/16/2019	Changed line spacing after section 6.9. Added database reference to 6.2. Removed SAU Chief from approval lines. Removed unit references to PMPs. Updated definition of certified reference material to comply with LOM.
7	07/15/2020	Removed fire debris from section 2 and section 3. Updated 5.1 for clarity.

Approval Redacted - Signatures on File

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QA Approval

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